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भाग 6 एक्स–रे प्रतिदीप्ति स्पेक्ट्रोमेट्रिक प्रणाली द्वारा टाईटेनियम का निर्धारण

Indian Standard

CHEMICAL ANALYSIS OF FERROTITANIUM

PART 6 DETERMINATION OF TITANIUM BY X-RAY FLUORESCENCE SPECTROMETRIC METHOD

ICS 77.100; 77.120.50

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

February 1998 **Price Group 1**

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 6) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' covers the chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard the Sectional Committee decided that a separate series for each ferroalloy be prepared. Accordingly, IS 1559 in its various parts was published for the methods of chemical analysis of ferrosilicon.

The methods of chemical analysis of various constituents of ferromolybdenum and ferrochromium are covered in a series of parts of IS 12614 and 13452 respectively. The chemical analysis of ferrotitianium is covered in a series of parts of this standard. With the publication of these parts the analysis of relevant constituent prescribed for ferrotitanium in IS 1559:1967 will be superseded. This part covers the method for determination of titanium by X-ray fluorescence spectrometric method. The other parts of this series are:

Part 1	Determination of carbon by the direct combustion (gravimetric) method
Part 2	Determination of silicon by gravimetric method
Part 3	Determination of titanium by cupferron (gravimetric) method
Part 4	Determination of aluminium
Part 5	Determination of sulphur

During this revision the latest method (XRF) for determination of titanium has been introduced.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard
CHEMICAL ANALYSIS OF FERROTITANIUM

PART 6 DETERMINATION OF TITANIUM BY X-RAY FLUORESCENCE SPECTROMETRIC METHOD

1 SCOPE

This standard (Part 6) describes the method for the determination of titanium in ferrotitanium by wavelength dispersive X-ray fluorescence (XRF) technique. It is applicable for titanium in the range from 20 to 75 percent.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070:1992	Reagent grade water (*third revision*)
1472:1977	Methods of sampling of ferroalloys for determination of chemical composition (*first revision)*

3 SAMPLING

The sample shall be drawn and prepared as per the method described in IS 1472.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF TITANIUM

5.1 Outline of the Method

The sample of ferrotitanium is fused with lithium metaborate and made into solid button. The plain surface of the button is exposed to primary X-rays. The secondary fluorescent radiation from the sample is dispersed and detected. The concentration, of titanium is obtained by relating to the titanium fluorescent X-ray intensity from the unknown sample to that of the standard reference material of known composition.

5.2 Equipments and Accessories

The different equipments that are required are pulverizer, crucibles and casting dish/moulds, muffle furnace and wavelength dispersive XRF spectrometer.

5.2.1 *Pulverizer*

It consists of a grinding box that can be set into vibrations of high frequency so that the pieces of samples placed in it can be powdered to 200 mesh size in 3 to 4 minutes.

5.2.2 *Crucibles and Casting Dish Moulds*

The crucibles and casting dish moulds made of a special alloy of 85 percent Platinum, 10 percent Rhodium and 5 percent gold may be used for the preparation of beads.

5.2.3 *Muffle Furnace*

An electric furnace capable of achieving a temperature of 1 200°C for a desired period of time must be used.

5.2.4 *Wavelength Dispersive X-ray Fluorescence Spectrometer*

It consists of an X-ray tube, a spectrometer chamber, a sample chamber, a mechanical arm, monochromators, a computer and associated electronic circuits. The X-ray tube acts as a source of primary X-rays. The spectrometer chamber houses the X-ray tube and monochromator. The monochromator consists of collimators to make the fluorescent X-rays a parallel beam filters to reduce background radiation, crystal to serve as the dispersion medium and detector for measuring the intensities. The sample chamber is separtated by means of a movable shutter from the spectrometer chamber. The sample can be introduced into a sample chamber by means of a mechanical arm. The computer and the associated electronic circuits interact with the different parts of the spectrometer to control the sequence of operations and also carry out the mathematical computations to display/print out the final result in terms of percentage.

6 STANDARDS AND REAGENTS

6.1 Standards

Certified reference materials of ferrotitanium of known composition as near in composition as the sample is to be analysed, shall be used for preparing the calibration curve.

6.2 Setting Up the Instrument

Two ferro-titanium standards/ samples, one containing low and the other containing high content of titanium should be used.

6.2.1 *Drift Correction*

The intensity from the two standards/samples as described in 6.2 may be stored in the computer alongwith the calibration curve. Before analysing the unknown samples, it is necessary to update the intensities from the standards to take care of any drift in the spectrometer.

6.3 Reagents

6.3.1 Only high purity anhydrous chemicals such as magnesium oxide, boric acid, lithium tetraborate and lithium iodide should be used.

7 SAMPLE PREPARATION

7.1 Samples may be prepared by fusion or by pallatization with binders. Different recipies are available for sample preparation. One method using lithium tetraborate and boric acid is given below.

Weigh 0.2 g of ferrotitanium and evenly spread it over a bed of 1 g magnesium oxide placed inside a platinum lid. Keep it inside a muffle furnace at 5 000°C for 30 minutes for oxidation. During this period, arrange a free flow of oxygen into the furnace to ensure complete oxidation.

After removing from the furnace allow the lid and its contents to cool in a desiccator. Transfer quantitatively the contents of the lid to a Pt-Rh-Au crucible. Add 8.0 g of anhydrous lithium tetraborate (40 times the weight of the sample), 0.4 g of boric acid and 0.2 g of lithium iodide. Mix the contents of the crucible well and place the crucible in muffle furnace at 1 200°C. Cover the crucible with Pt-Rh-Au lid and allow it to heat for 10 minutes. Take out the crucible with a pair of tongs and rotate the crucible for thorough mixing and keep it again in the furnace. After 10 minutes, remove the lid and place it on an even platform and transfer the fused melt from the crucible to the lid and allow it to cool. When the temperature of the bead comes to that of the surroundings, remove it from the lid. It is now ready for irradiation.

8 CALIBRATION AND ANALYSIS

8.1 Prepare the fused beads of ferrotitanium of certified standard reference materials of known composition following the procedure specified in **7**.

NOTE — Take care that the ferrotitanium standards selected must be within ± 10 percent of titanium content of the sample to be analysed.

8.2 These beads are then irradiated. The 'K' X-rays of titanium from these beads may be monitored by using a wavelength dispersive, XRF spectrometer described in **5.2.4**. Select the crystal, detector and the X-ray line listed in Table 1. Each of these beads must be irradiated at least 3 times and the average value of the intensity detected must be used for calibration. These intensities must be correlated to their concentrations and the calibration curve thus obtained may be stored in the computer. The concentration in the case of unknown samples can be obtained by referring to this curve.

Table 1 Selection of Parameters

(*Clause* 8.2)

Element	X-ray Line Selected	Wavelength °A	Crystal	Detector
Titanium	K	2.750	LiF	Any suitable detector

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